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IS 6947-1 (1973): Methods of estimation of composite pigments in oil pastes, ready mixed paints and enamels, Part 1: Estimation of lead, zinc oxide, titanium dioxide, calcium compounds and zinc sulphide [CHD 20: Paints, Varnishes and Related Products]

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“Knowledge is such a treasure which cannot be stolen”



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IS : 6947 (Part I) 1973

Indian Standard

**METHODS OF ESTIMATION OF
COMPOSITE PIGMENTS IN OIL PASTES
AND READY MIXED PAINTS**

**PART I ESTIMATION OF LEAD, ZINC OXIDE,
TITANIUM DIOXIDE, CALCIUM COMPOUNDS
AND ZINC SULPHIDE**

(First Reprint MAY 1984)

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AMENDMENT NO. 1 JUNE 1975
TO

IS:6947(PART I) - 1973 METHODS OF ESTIMATION
OF COMPOSITE PIGMENTS IN OIL PASTES AND
READY MIXED PAINTS

Part I Estimation of Lead, Zinc Oxide,
Titanium Dioxide, Calcium Compounds
and Zinc Sulphide

A l t e r a t i o n s

(First cover page and pages 1 and 3,
title) - Substitute the following for the
existing title:

'Indian Standard
METHODS OF ESTIMATION OF COMPOSITE
PIGMENTS IN OIL PASTES, READY
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Part I Estimation of Lead, Zinc Oxide,
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and Zinc Sulphide'

(Pages 3 and 4, clauses 0.3 and 1.1,
last line; and clause 4.1, first line) - Substitute
'ready mixed paints and enamels' for
'ready mixed paints' at all the places.

(CDC 50)

Reprography Unit, ISI, New Delhi

Indian Standard

METHODS OF ESTIMATION OF COMPOSITE PIGMENTS IN OIL PASTES AND READY MIXED PAINTS

PART I ESTIMATION OF LEAD, ZINC OXIDE, TITANIUM DIOXIDE, CALCIUM COMPOUNDS AND ZINC SULPHIDE

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Indian Standard

METHODS OF ESTIMATION OF COMPOSITE PIGMENTS IN OIL PASTES AND READY MIXED PAINTS

PART I ESTIMATION OF LEAD, ZINC OXIDE, TITANIUM DIOXIDE, CALCIUM COMPOUNDS AND ZINC SULPHIDE

0. FOREWORD

0.1 This Indian Standard (Part I) was adopted by the Indian Standards Institution on 5 March 1973, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

0.2 While finalizing IS : 101-1964* for publication, the concerned Sectional Committee was confronted with the problem whether to include standard methods of tests for single pigments or not. It was decided that under clause 27.1.1.1 of IS: 101-1964* it may be clearly indicated that analysis of single pigments shall be done as specified in individual standard specifications and analysis of composite pigments in accordance with a separate standard. Consequently, formulation of this standard was taken up. The Committee, however, agreed to phase the work and to issue in parts, the methods of estimation of composite pigments which would comprise such methods as have been developed and standardized. This part covers the methods of estimation of lead, zinc oxide, titanium dioxide, calcium compounds and zinc sulphide. The other parts would include estimation of lead chromes, zinc chromes, lead sulphate, Prussian blue, ferric oxide, aluminium, etc. It is hoped that these standards would prove of great assistance to paint technologists engaged in testing of paints and allied materials.

0.3 This standard for method of test is a necessary adjunct to material specification for oil pastes and ready mixed paints.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

*Methods of test for ready mixed paints and enamels (*second revision*).

†Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard (Part I) prescribes methods of estimation of lead, zinc oxide, titanium dioxide, calcium compounds and zinc sulphide in composite pigments of oil pastes and ready mixed paints.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in 2 of IS: 101-1964* and IS: 1303-1963† shall apply.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (*see IS : 1070-1960‡*) shall be employed in the tests.

NOTE—'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. EXTRACTION OF PIGMENTS

4.1 Pigments from oil pastes and ready mixed paints shall be first extracted by the procedure given in 27 of IS: 101-1964*.

5. DETERMINATION OF TOTAL LEAD

5.1 Reagents

5.1.1 *Dilute Sulphuric Acid— 1:20 (v/v).*

5.1.2 *Ammonium Acetate Solution — 20 percent (m/v).*

5.1.3 *Acetic Acid— 1 : 2 (v/v).*

5.1.4 *Ammonium or Sodium Tartrate Solution — 10 percent (m/v).*

5.1.5 *Potassium Chromate Solution — 4 percent (m/v).*

5.1.6 *Sodium Acetate Solution — 1 percent (m/v).*

5.1.7 *Dilute Hydrochloric Acid — 1:1 (v/v).*

5.1.8 *Potassium Iodide— solid.*

5.1.9 *Standard Sodium Thiosulphate Solution — 0.1 N (*see 32 of IS : 2316-1968§*).*

*Methods of test for ready mixed paints and enamels (*second revision*).

†Glossary of terms relating to paints (*revised*).

‡Specification for water, distilled quality (*revised*).

§Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision*).

5.1.10 Starch Solution — Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into 1 litre of water. Boil for three minutes. Allow to cool and decant off the supernatant clear liquid.

5.2 Procedure

5.2.1 Weigh accurately 0.5 to 1 g of the isolated pigment in a dry 250-ml beaker and incinerate on a low flame over wire gauge to destroy organic matter particularly in case of prussian blue. Cool, add 60 to 70 ml dilute sulphuric acid and break up the agglomerated particles by stirring with a glass rod. Boil for 2 to 3 minutes and wash down the sides of the beaker with 25 to 30 ml of water. Cool the beaker for 1 hour in ice-cold water or allow it to stand for overnight. Filter the residue through a sintered glass crucible (G. No. 4) or a Cooch crucible and wash the residue twice with 10 ml of cold water. Preserve the filtrate for the estimation of zinc oxide.

5.2.2 Wash this residue 5 times under slow suction with hot ammonium acetate solution taking 10 ml each time. Wash it again twice with 5 ml of acetic acid and then twice or thrice with hot water. Transfer the filtrate into the beaker and add 10 ml of ammonium or sodium tartarate solution. Make up the volume of the solution to about 150 ml with water and boil the solution. Add drop by drop 10 ml of potassium chromate solution and stir constantly with a glass rod. Boil for 5 to 10 minutes and then place the beaker on a water-bath for 15 to 30 minutes. Filter the lead chromate precipitate through a sintered glass crucible (G. No. 4) and wash with 1 percent sodium acetate solution till it is free from chromate ions.

5.2.3 Cool and dissolve lead chromate precipitate in about 20 ml of dilute hydrochloric acid (this 20 ml of hydrochloric acid solution shall be added in three instalments of 10, 5 and 5 ml) under slow suction. Wash the precipitate 3 to 4 times with cold water. Transfer the solution in the same beaker and adjust the volume with water to about 150 to 200 ml. Add 2 g of potassium iodide and titrate the liberated iodine with Standard sodium thiosulphate solution using 2 ml of starch solution.

NOTE— Lead chromate precipitate may also be filtered over a small filter paper (Whatman No. 30), and the precipitate along with the filter paper transferred to the beaker and treated with 20 ml of hydrochloric acid solution for titration.

5.3 Calculation

$$\text{Total lead, as lead monoxide } (\text{PbO}), \text{ percent by mass} = \frac{7.43 \times V \times N}{M}$$

where

V = volume in ml of standard sodium thiosulphate solution used,

N = normality of standard sodium thiosulphate solution, and

M = mass in g of the material taken for the analysis.

6. DETERMINATION OF ZINC OXIDE

6.1 Reagents

6.1.1 Ammonium Hydroxide Solution — 1:1 (*v/v*).

6.1.2 Standard Potassium Ferricyanide Solution — 0.1 N. This solution is prepared in cold water, filtered rapidly and kept in a dark colour bottle. The solution, if kept in dark, may be used for seven days.

6.1.3 Standard Sodium Thiosulphate Solution — 0.1 N (*see 32 of IS : 2316-1968**).

6.1.4 Starch Solution — same as in **5.1.10**.

6.1.5 Ammonium Bifluoride — solid.

6.1.6 Potassium Iodide — solid.

6.2 Procedure — Take the filtrate preserved in **5.2.1** in a 500-ml conical flask and add ammonium hydroxide solution drop by drop until the solution is just alkaline. (An excess of not more than 3 to 4 drops of ammonium hydroxide solution should be added as it is essential that the blue litmus should turn red with ammonium bifluoride, to be added subsequently.) Dissolve the ferric hydroxide, precipitate if any, by adding ammonium bifluoride in small amounts. Cool the solution, add 2 to 3 g of ammonium bifluoride and 3 g of potassium iodide. Dilute with water to make up the volume to approximately 250 ml. Add about 2 ml of starch solution and titrate the liberated iodine, if any, within 30 seconds with standard sodium thiosulphate solution. Then add standard potassium ferricyanide solution depending on the amount of zinc oxide present, in accordance with Table 1, such that the excess ferricyanide solution shall not exceed 5 ml. Shake the flask, keep it in dark for 2 to 3 minutes and then titrate the liberated iodine against standard sodium thiosulphate solution.

NOTE — The end point is very sharp. At the end point the colour will be bright pale greenish-yellow. If there is no such colour except whitish precipitate, a further quantity of 3 to 4 ml of ferricyanide solution should be added and the liberated iodine titrated with standard thiosulphate solution. The results will not be affected by this further addition of ferricyanide solution in 1 or 2 instalments.

*Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision*).

TABLE I AMOUNT OF POTASSIUM FERRICYANIDE SOLUTION TO BE ADDED

(Clause 6.2)

PROBABLE AMOUNT OF ZINC OXIDE IN THE MATERIAL	AMOUNT OF MATERIAL TO BE TAKEN FOR THE EXPERIMENT	VOLUME OF STANDARD POTASSIUM FERRICYANIDE SOLUTION TO BE ADDED	FINAL VOLUME OF THE SOLUTION TO BE ADJUSTED	AMOUNT OF ZINC OXIDE COVERED BY THE ADDITION OF STANDARD POTASSIUM FERRICYANIDE SOLUTION
(1) percent by mass	(2) g	(3) ml	(4) ml	(5) percent by mass
0.5 to 3	1.0	3 to 3.5	150	0.5 to 4
5	1.0	7.5	250	3.1 „ 9
8	1.0	10.0	„	6.2 „ 12
10	1.0	11.0	„	7.4 „ 13
15	1.0	15.0	„	12.4 „ 18
20	1.0	18.0	„	16.2 to 22
20	0.5	11.0	„	15 „ 27
25	1.0	22.5	„	22 „ 27
25	0.5	13.0	„	20 „ 32
30	1.0	26.5	„	26.8 „ 32.5
30	0.5	14.0	„	22.5 to 34.5
33	1.0	28.5	„	29.3 „ 35
33	0.5	15.0	„	25 „ 37
50	0.5	22.5	„	44 „ 55.5
100	0.4	35.0	„	93.5 „ 100

6.2.1 In case of low contents of zinc oxide (5 to 20 mg), make up the volume of the test solution to 125 to 150 ml, add 2 g of potassium iodide, 2.5 to 3.5 ml of standard potassium ferricyanide solution and then titrate with standard sodium thiosulphate solution, preferably from a micro-burette.

6.3 Calculation

$$\text{Zinc oxide, percent by mass} = \frac{12.45 \times V \times N}{M}$$

where

V = volume in ml of standard sodium thiosulphate solution used,

N = normality of standard sodium thiosulphate solution, and

M = mass in g of the pigment taken for the analysis.

7. DETERMINATION OF TITANIUM DIOXIDE

7.1 Reagents

- 7.1.1 *Dilute Hydrochloric Acid* — 1:1 (*v/v*).
- 7.1.2 *Concentrated Sulphuric Acid*— conforming to IS:266-1961*.
- 7.1.3 *Ammonium Persulphate*— solid.
- 7.1.4 *Concentrated Nitric Acid*— conforming to IS:264-1968†.
- 7.1.5 *Dilute Sulphuric Acid* — 1:10 (*v/v*).
- 7.1.6 *Hydrogen Peroxide*— conforming to IS:2080-1962‡.
- 7.1.7 *Standard Titanium Dioxide Solution* — Weigh exactly 1.00 g of pure calcined titanium dioxide, previously dried at 110°C, in a platinum crucible, with 10 g of sodium pyrosulphate. Raise the temperature to fusion point until the liquid no longer contains solid particles of titanium dioxide. Cool the fused mass, dissolve in cold dilute sulphuric acid, filter, make up the solution to 500 ml with dilute sulphuric acid of the same strength and mix thoroughly. Take 10 ml of the solution in a 100-ml measuring flask and dilute with water up to the mark. One millilitre of this solution is equivalent to 0.000 2 g of titanium dioxide.

7.2 Procedure

7.2.1 Weigh accurately about 0.5 g of the isolated pigment in a 250-ml beaker. Add 50 ml of dilute hydrochloric acid and boil gently for about 5 minutes. Dilute the solution to about 200 ml with water and boil again for 2 minutes. Place the beaker on a water-bath and boil for about 2 to 3 hours. Filter the hot solution through a filter paper (Whatman No. 30) and wash the residue with hot water till the filtrate is free from chloride and lead ions. Preserve this filtrate for the determination of calcium compounds.

7.2.2 Place the filter paper along with the residue in an air-oven for about half an hour to remove moisture (complete drying is not necessary). Transfer the filter paper with residue in a 400-ml dry beaker, add 20 ml of concentrated sulphuric acid, 3 to 4 g of ammonium persulphate and disperse the filter paper and residue with a glass rod. Wash down the glass rod with 10 ml of concentrated nitric acid. Cover the beaker with a watch-glass and fume strongly for about 3 to 4 hours. Cool and dilute to about 200 ml with dilute sulphuric acid. Boil the solution for about 2 to 3 minutes and allow to stand on the water-bath for

*Specification for sulphuric acid (*revised*).

†Specification for nitric acid (*first revision*).

‡Specification for stabilized hydrogen peroxide.

about 2 hours. Filter the solution through a filter paper (Whatman No. 30) in a 1 000-ml measuring flask and wash the residue thoroughly with hot dilute sulphuric acid. Cool the filtrate and make up to the mark with water. Take an aliquot portion and determine titanium dioxide colorimetrically with hydrogen peroxide. (A spectro-photometer may also be used for colorimetric determination.)

NOTE — If the insoluble residue is not baked or ignited, then fuming once with sulphuric acid as described above shall bring all the titanium dioxide in solution in most cases. The residue obtained after fuming should be preserved for checking of titanium dioxide, if required.

7.3 Calculation

$$\text{Titanium dioxide, percent by mass} = \frac{0.02 V}{M}$$

where

V = volume in ml of dilute standard titanium dioxide solution required to match the colour, and

M = mass in g of the pigment present in the aliquot portion taken for the test.

8. DETERMINATION OF CALCIUM COMPOUNDS

8.1 Reagents

8.1.1 *Dilute Sulphuric Acid* — 1:4 (v/v).

8.1.2 *Ammonium Hydroxide* — 20 percent.

8.1.3 *Hydrogen Sulphide* — gas.

8.1.4 *Ammonium Oxalate Solution* — saturated.

8.1.5 *Standard Potassium Permanganate Solution* — 0.1 N (see 27 of IS : 2316-1968*).

8.2 Procedure

8.2.1 Treat the filtrate obtained in 7.2.1 with a few drops of, dilute sulphuric acid and boil. Make the solution alkaline to litmus with ammonium hydroxide and pass a stream of hydrogen sulphide gas through this solution for 15 minutes. Filter off the sulphides and wash 2 to 3 times with saturated solution of hydrogen sulphide. Boil the filtrate to remove hydrogen sulphide and continue boiling after acidifying the solution. Make the solution alkaline to litmus with ammonium hydroxide and heat to boil. Add sufficient quantity of ammonium oxalate

*Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision*).

solution and boil for 5 minutes. Allow the beaker with the precipitate to stand for 2 hours on a water-bath. Filter the residue through a filter paper (Whatman No. 30) and wash several times with hot water. Ignite it in a porcelain crucible to calcium oxide, cool in a desiccator and weigh to a constant mass.

8.2.2 Alternatively, the residue on the filter paper be washed with hot water until the filtrate is free from oxalate ions. Wash down the residue on the filter paper into the original beaker with hot water and wash the filter paper wash dilute sulphuric and collecting the washings in the beaker. Heat to about 70 to 80°C and titrate with standard potassium permanganate solution. When the end point is reached, the filter paper from which the precipitate was washed out shall be put in the beaker and the titration concluded by adding further quantity of permanganate solution. (For filter paper not more than two drops of standard potassium permanganate need be required.)

8.3 Calculations

8.3.1 Ignition Method

$$\text{Calcium oxide, percent by mass} = 100 \times \frac{M_1}{M_2}$$

where

M_1 = mass in g of calcium oxide obtained, and

M_2 = mass in g of the pigment taken for the analysis.

8.3.2 Titration Method

$$\text{Calcium oxide, percent by mass} = \frac{0.28 VN}{M_2}$$

where

V = volume in ml of standard potassium permanganate solution,

N = normality of potassium permanganate solution, and

M_2 = mass in g of the pigment taken for the analysis.

9. DETERMINATION OF ZINC SULPHIDE AND ZINC OXIDE

9.1 Reagents

9.1.1 Dilute Acetic Acid — 3 percent (v/v).

9.1.2 Concentrated Hydrochloric Acid — conforming to IS : 265-1962*.

9.1.3 Perhydrol or Concentrated Nitric Acid — conforming to IS : 264-1968†.

9.1.4 Dilute Sulphuric Acid — 1:20 (v/v).

*Specification for hydrochloric acid (*revised*).

†Specification for nitric acid (*first revision*).

9.2 Procedure—Treat 1 g of the isolated pigment with 100 ml of dilute acetic acid, stir well with a glass rod for 5 minutes and allow it to stand for overnight. Filter and wash the residue with dilute acetic acid. Determine zinc oxide content of the filtrate iodometrically as prescribed in 6.2. Transfer the residue in a beaker, add 20 to 25 ml of concentrated hydrochloric acid and boil off hydrogen sulphide. Add few drops of concentrated nitric acid or perhydrol cautiously, boil for few minutes and evaporate nearly to dryness on a water-bath. Add 1 ml of concentrated hydrochloric acid and 5 ml of distilled water, and again evaporate to dryness on a water-bath. Treat the residue with 50 ml of dilute sulphuric acid and filter, if necessary. Determine zinc sulphide by titrating the zinc oxide content of this solution as prescribed in 6.2.

9.3 Calculation

$$\text{Zinc sulphide, percent by mass} = \text{Zn O} \times 1.197 \times 100$$

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Panel for Organic Pigments, CDC 50 : 1 : 2

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>
Length	metre	m
Mass	kilogram	Kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>	<i>Definition</i>
Force	newton	N	$1 \text{ N} = 1 \text{ kg.m/s}^3$
Energy	joule	J	$1 \text{ J} = 1 \text{ N.m}$
Power	watt	W	$1 \text{ W} = 1 \text{ J/s}$
Flux	weber	Wb	$1 \text{ Wb} = 1 \text{ V.s}$
Flux density	tesla	T	$1 \text{ T} = 1 \text{ Wb/m}^2$
Frequency	hertz	Hz	$1 \text{ Hz} = 1 \text{ c/s (s}^{-1}\text{)}$
Electric conductance	siemens	S	$1 \text{ S} = 1 \text{ A/V}$
Electromotive force	volt	V	$1 \text{ V} = 1 \text{ W/A}$
Pressure, stress	pascal	Pa	$1 \text{ Pa} = 1 \text{ N/m}^2$

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22E Kalpana Area	BHUBANESHWAR 751014	5 36 27
5-8-56C L.N. Gupta Marg	HYDERABAD 500001	22 10 83
R 14 Yudhister Marg, C Scheme	JAIPUR 302005	6 98 32
117/418 B Sarvodaya Nagar	KANPUR 208005	4 72 92
Patliputra Industrial Estate	PATNA 800013	6 28 08
Hantex Bldg (2nd Floor), Rly Station Road	TRIVANDRUM 695001	32 27

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